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(54) Beam-diverging multi-aperture diffraction plate

(57) A membrane 14 increases the divergence of a beam of electromagnetic radiation by means of diffraction via a multiplicity of apertures 16 each of which is comparable in size to the wavelength of the electromagnetic radiation whose divergence is being altered. Its function is similar to that of a ground glass diffuser in visible light, except that the increased divergence is caused by diffraction rather than refraction. It is of particular use, though not limited to, regions of the electromagnetic spectrum where refraction optics are difficult or costly to fabricate, such as the well known soft x-ray regime from 0.5 to 50 nm..

In one embodiment the membrane 14 is opaque and has a multiplicity of small apertures 16. These apertures 16 are chosen to be of the appropriate size with respect to the wavelength of the light being used so that the incident beam of collimated radiation 10 is diffracted into a diffuse cone of radiation 12. The required aperture 16 size can be related to the wavelength of the light being used and the semi-angle of the cone of diffraction required by the well known diffraction formulae. Alternatively the membrane may be transparent and be provided with a multiplicity of appropriately sized opaque particles or regions having a different phase charge characteristic.

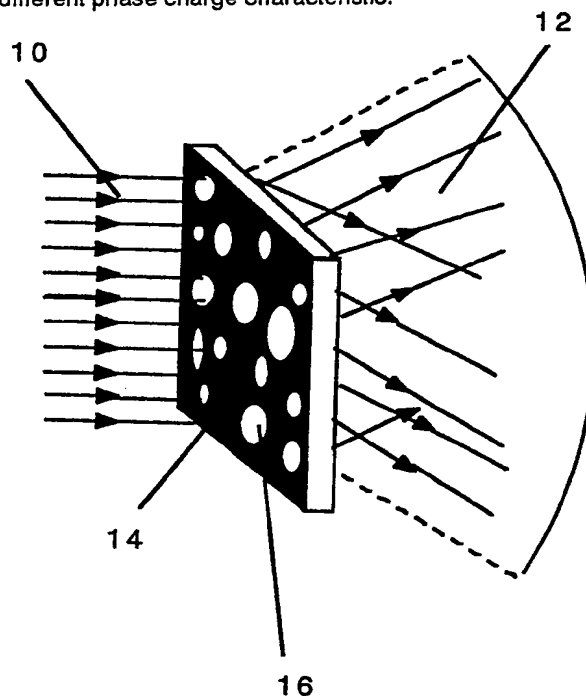


Fig. 1

The claims were filed later than the filing date within the period prescribed by Rule 25(1) of the Patents Rules 1990.

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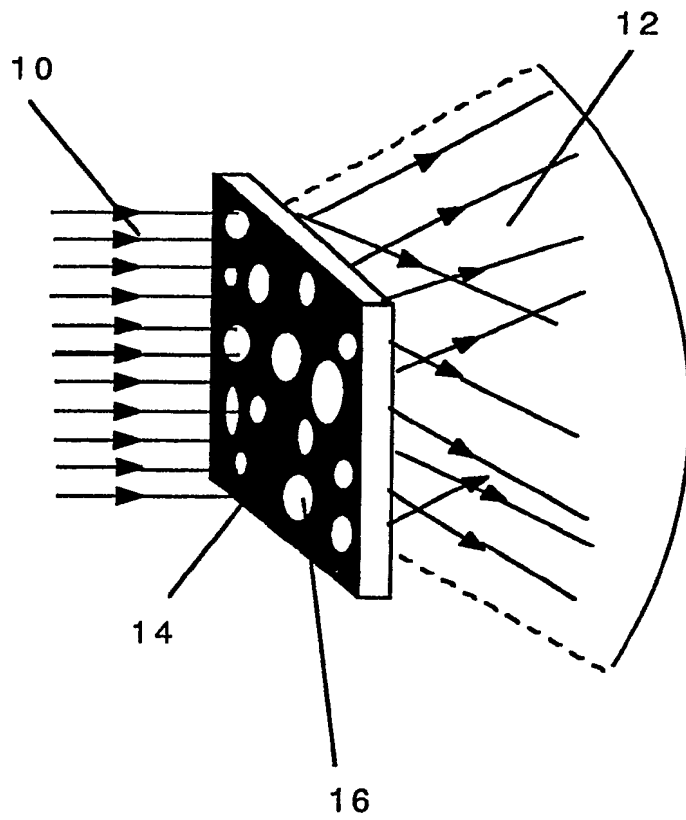


Fig. 1

2 of 4

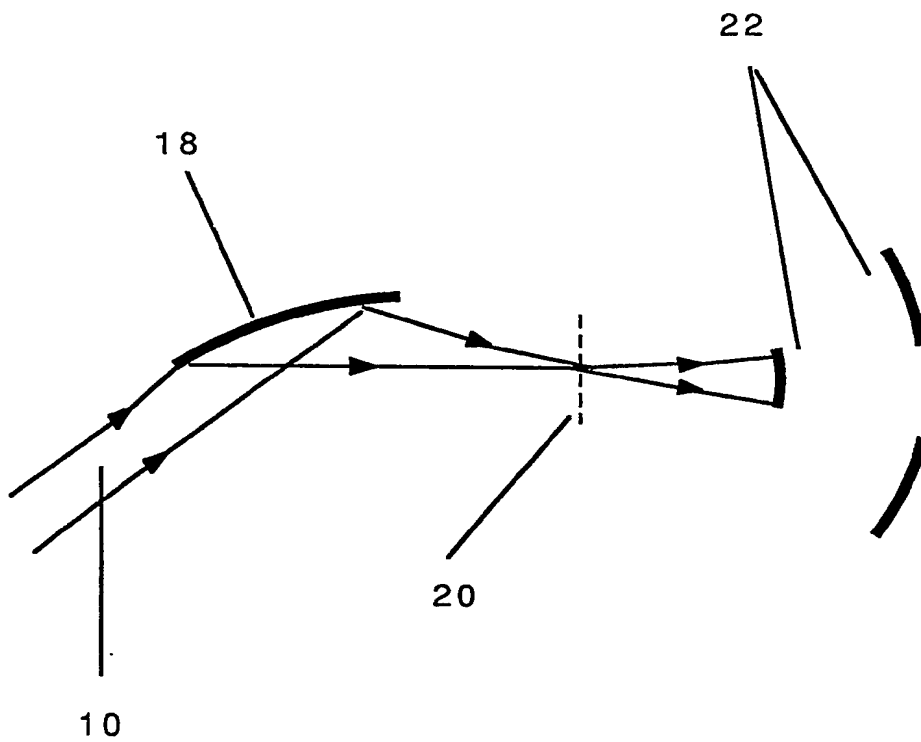


Fig. 2

3 of 4

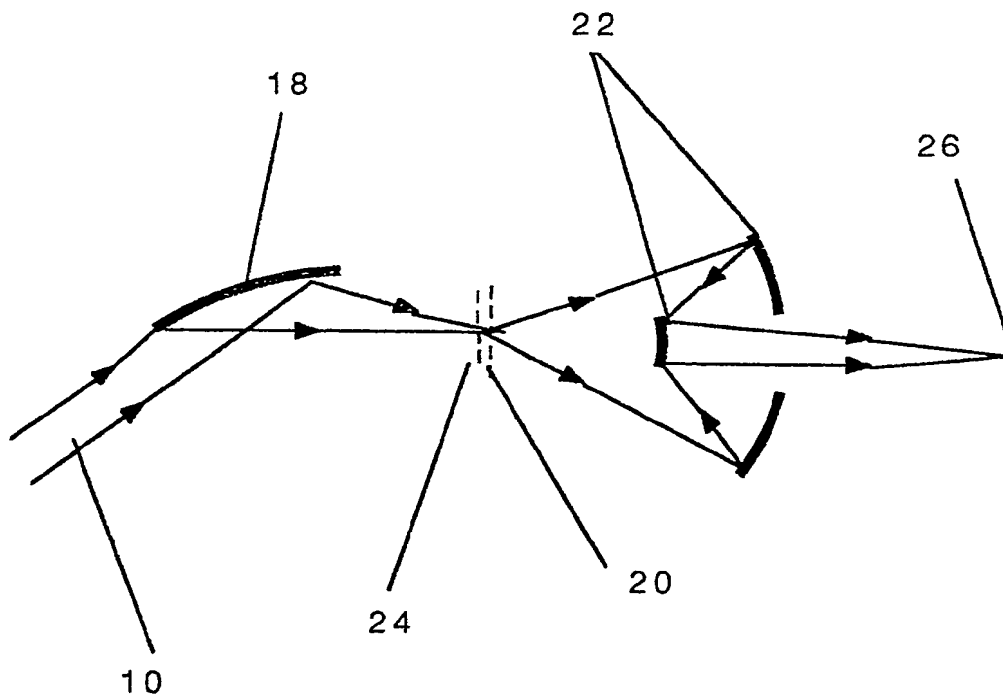


Fig. 3

4 of 4

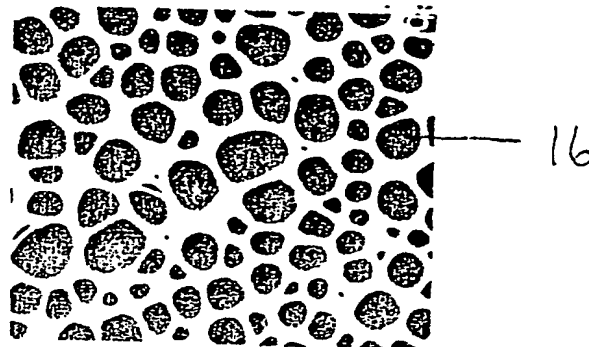


Fig. 4

2 μ m

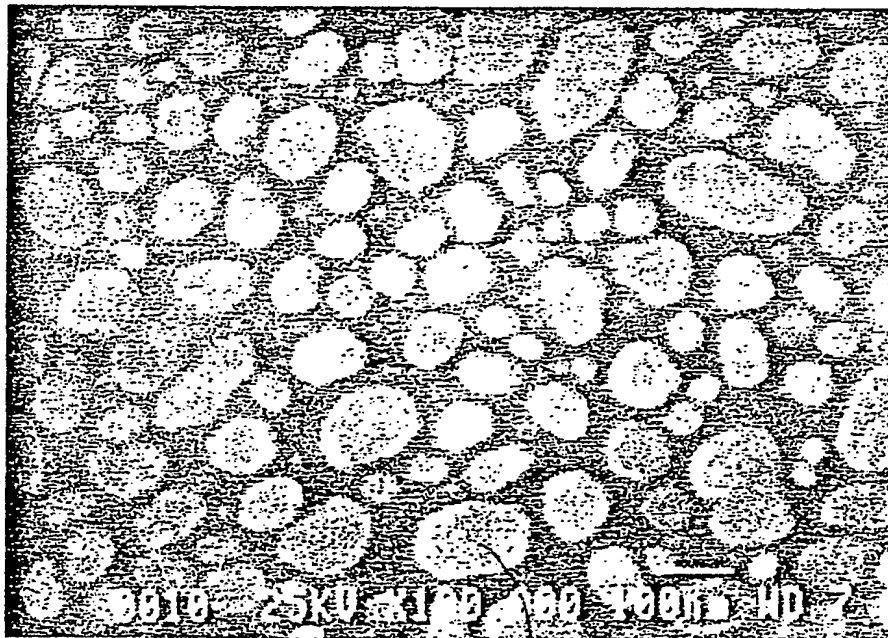


Fig. 5

BEAM-DIVERGING MULTI-APERTURE DIFFRACTION PLATE

This invention concerns a beam expanding diffraction optic especially constructed to increase the divergence, or effective numerical aperture, of an electromagnetic beam. It is of particular use in regions of the electromagnetic spectrum where refraction optics are difficult or costly to fabricate, such as the soft x-ray regime. Particular applications include matching the numerical aperture of the illuminating source of a microscope to that of the imaging optics for optimum resolving power.

In one version of the device, it comprises a suitable opaque membrane with a large number of apertures. Each of the apertures is of an appropriate size so as to diffract a portion of an incoming beam of electro magnetic radiation into the required angle of divergence, as illustrated schematically in Fig. 1. Its function is similar to that of a ground glass screen in visible light, except that the increased divergence is caused by diffraction rather than refraction.

One use for such a device is in the field of soft x-ray imaging microscopy.

Soft x-ray microscopy has long been advocated as a significant step forward in viewing hydrated biological material. Thanks to the development of suitable optics for this wavelength region over the past decade, experiments at synchrotron sources and large x-ray lasers have begun to demonstrate the potential advantages, i.e. that hydrated, unstained, thick (around 1 μm) specimens can be seen at resolutions of 0.05 μm . This is an order of magnitude better than is possible with conventional optical microscopy, and approaching the resolution of routine transmission electron microscopy (TEM). It is a very promising complementary technique, as TEM specimens are dehydrated and metal stained in order to achieve the desired contrast. X-ray optics currently being developed will allow the resolution to reach 10 to 20 nm.

The optical components of a transmission imaging x-ray microscope are very similar to a conventional visible light microscope. They both require a source of illumination, a condenser that focuses the light onto the specimen being examined and an imaging lens that forms a magnified image, either for the eye to see or onto film or other recording medium.

At x-ray wavelengths refraction is negligible so conventional lenses don't work. They have to be replaced either by zoneplates, which are well known devices

that operate by diffraction, or by mirrors with well known special multilayer coatings to enable them to reflect reasonably (10 to 30 % per surface). Both zoneplates and multilayer coated mirrors have one common peculiarity when used in imaging - they require a central obstruction. This means they have both a maximum and a minimum cone of radiation they can accept. In zoneplates, this is a result of the need to block the zero order radiation, creating a shadow in which the image is visible. In mirror systems it is because of the use of the well known Schwarzschild two mirror design. This arrangement allows the correction of the three primary aberrations, spherical, coma and astigmatism, over a reasonable field with only two spherical surfaces. In soft x-ray imaging the condenser now serves two purposes - it increases the flux through the specimen, and it irradiates it with rays that will fill the entire aperture of the imaging optic. This second function is important, as unless the condenser cone of radiation matches that of the imaging optic, only a minute amount of light diffracted by the very smallest features will pass through the whole system. The low resolution features of the image, contained in less diffracted light, will not be seen. The result would be an extremely faint image containing only the outline of the smallest features and sharp edges. It would make focussing almost impossible and would be difficult to interpret. By matching the cones of radiation (i.e. the numerical apertures), all aspects of the image will be visible, from the smallest detail to the low resolution parts of the specimen. The need to match the numerical aperture of the condenser optics to that of the imaging optic results in fabrication difficulties. In the case of zoneplates, where the numerical aperture is determined by the width of the outer most zone, a condenser that is larger than the imaging zoneplate and so collects more light, is considerably more difficult to fabricate than a micro zoneplate of the same numerical aperture. In the case of Schwarzschild mirrors, the condenser and imaging optic require about the same effort.

Grazing incidence optics are easier to fabricate than normal incidence multilayer optics, and more efficient in terms of percentage of flux transferred. They can be used to focus the x-ray laser source to an area a few tens of μm in diameter. Their major drawback as imaging optics is the severe aberrations caused by the extreme off axis design. Whilst this is a problem in imaging, in the condenser of a microscope aberrations are essentially irrelevant. As condensers the problem is that the limited angles of high efficiency reflection give a maximum cone of illumination of a few degrees (numerical aperture ~ 0.05), under filling the imaging zone-plates or Schwarzschild mirrors, which require a cone of illumination of at least ± 6 degrees ($\text{NA} = 0.1$) for 100 nm imaging at 18.2 nm. (For 50 nm resolution, the NA required at 18.2 nm is 0.22) The difficulties of such a mismatch are illustrated in Fig.2. However, by combining a grazing incidence ellipsoid and the diffraction plate, as shown in Fig. 3, the numerical apertures can be matched, without introducing any further

alignment complications. It acts much like a ground glass screen in visible light, except that it works by diffraction from a large number of very aperture in an opaque film rather than scattering. A schematic drawing of a section of a diffraction plate in operation is shown in Fig. 1. The estimate is that such a system would transmit 40% of the 18.2 nm radiation originating from the source. A broad band multilayer coated optic that produced the same cone of illumination would only have a total transmission of about 4% (20% reflection per surface). The resultant order of magnitude increase in flux through the system will allow smaller power, less expensive x-ray laser sources to be used - an important consideration for commercialization.

A particular embodiment of the invention will now be described with particular reference to the attached figures in which:

Figure 1 is a schematic, isometric drawing of a small portion of a diffraction plate

Figure 2 is a schematic drawing of the optical components of an imaging soft x-ray microscope

Figure 3 is a schematic drawing of the optical components of an imaging soft x-ray microscope incorporating the soft x-ray diffusing diffraction plate.

Figure 4 is an electron micrograph of lacy formvar.

and

Figure 5 is an electron micrograph of the early stages of gold deposition.

A small portion of the beam-diverging multi-aperture diffraction plate (soft x-ray diffuser) 24, is shown in fig.1. It consists of an opaque membrane 14 in which there are a number of small apertures 16. These apertures 16 are chosen to be of the appropriate size with respect to the wavelength of the light being used so that the incident beam of collimated radiation 10 is diffracted into a diffuse cone of radiation 12. The required aperture 16 size can be related to the wavelength, λ , of the light being used and the semi-angle α of the cone of diffusion required, by the well known diffraction formula:

$$\text{diameter of aperture} = 0.4\lambda/\sin(\alpha) \quad \dots (1)$$

Although this formula is for the first minimum of diffraction at a straight edge, it is a good enough approximation. Lacy formvar, a well known transmission

electron microscopy support material provides just such a membrane 14 with appropriate sized apertures 16. As shown in the electron micrograph of Fig. 4. it is a web-like membrane, used to support thin sections too fragile to be mounted on a standard grid. It may be prepared by shaking an emulsion of formvar and glycerol, coating it as a thin film and then baking which perforates the film. By using an ultrasonic wand or bath, a much finer emulsion can be produced, with correspondingly finer perforations on baking. By controlling the size of the perforation, which are the apertures 16, the diffuser 24 can be tailored to wavelength and diffraction angle. After the films are made they may be coated with gold or other heavy metal in order to make the opaque regions non-transmitting to the required wavelength.

The need for the soft x-ray diffuser 24 is evident from fig. 2. The collimated soft x-ray radiation 10, from a synchrotron bending magnet, an undulator, a laser produced plasma, a soft x-ray laser, or similar source of soft x-ray radiation, is focussed onto a specimen 20 by a grazing incidence off axis ellipsoid 18. However, because of the limited grazing incidence angle which will reflect soft x-rays (for example the critical angle for gold coated surfaces at 18.2 nm is about 10 degrees) the maximum angle of the cone of radiation illuminating the specimen is considerable less than the 16 degrees required to fully illuminate a 0.28 numerical aperture Schwarzschild multilayer coated mirror, 22, required to give 50 nm resolution at 18.2 nm wavelength. As can be seen in fig. 2, the front mirror of the Schwarzschild imaging system 22 obscures the incoming beam, resulting in no or very little radiation passing through the system. However, by placing the diffraction soft x-ray diffuser 24 just before the specimen 22, as shown in Fig. 3, the cone of illumination now matches that required by the imaging mirrors 22. The result is flux through the system and a high resolution, high visibility image 26 being formed.

From equation 1, it can be seen that 25 nm diameter apertures would be required to diffract 18.2 nm wavelength light into a semi-angle of 21 degrees, or a numerical aperture of 0.36. This would be sufficient to illuminate optics capable of 30 nm resolution at 18.2 nm.

An alternative form of the diffracting plate, 24, would have a transparent film, with opaque particles of the appropriate size, whose diameter would also be given, to a good approximation, by equation 1. In depositing metallic material, especially gold, onto substrates by the well known techniques of evaporation or sputtering, the initial stages of thin film growth are characterized by nucleation of the coating material at random sites. As the procedure is continued these nucleation sites begin to coalesce and form complicated discontinuous patterns, as shown in Fig. 5. By stopping the deposition at the right time, an ensemble of randomly distributed diffracting objects of the appropriate size may be formed.

The holes or particle need not be uniform in shape, and it may well be an advantage to have a range of sizes to more uniformly fill the diffracted cone.

Both these schemes require blocking roughly 50% of the incident light. However, if the particles were not opaque, but of a material that introduced a half wavelength phase shift, the plate could be diffracting and transmit close to 100 % of the light.

Another method of construction would be to coat a thin film of either carbon or free standing aluminum with a heterodisperse (> 15% variability) monolayer of colloidal gold, with mean particle size chosen to be about 30 nm. At the appropriate concentration, about 50% of the light would be transmitted and diffracted into a cone with angles up to 25 degrees. (It may be better to use a homodisperse solution, with particles of 20 nm and rely on the maximum diffraction being at the edges, with lower diffraction away from the particle edges. This may also allow for a lower concentration of colloidal gold spheres to allow for greater than 50% throughput).

Lithography, including electron beam, uv, visible and x-ray lithography, is a well known technique which allows the production of complicated patterns on a rigid membrane at high resolution, and could be used for production of either type of diffraction plate.

Aerogels are well known gels in which liquid has been replaced by air. Such solids are characterized by extremely high porosity and concomitantly low density. Aerogels have been formed from a wide variety of substances including metals such as tungsten. Investigation shows that they are made up of dense clusters which form small spheres. These spheres connect in a space filling chain-matrix with lengths from 2 to 50 nm. Diffusion plates may be made from a thin microtomed section of the bulk material.

The diffraction plate could be used for non- electromagnetic radiation. With apertures or particles of the appropriate size, the principle could be used for neutron or electron diffraction, using their well known DeBroglie wavelength. It could also be used to diffuse sound waves or similar.

BEAM-DIVERGING MULTI-APERTURE DIFFRACTION PLATE

CLAIMS

- 1 An optic for increasing the divergence of a beam of electromagnetic radiation by means of diffraction via a multiplicity of apertures each of which is comparable in size to the wavelength of the electromagnetic radiation whose divergence is being altered.
- 2 An optic as claimed in Claim 1 made by having a membrane that is substantially opaque to the electromagnetic radiation of interest through which holes of suitable dimension have been produced.
- 3 An optic as claimed in Claim 1 made by having a membrane that is substantially transparent to the electromagnetic radiation of interest onto or into which have been introduced a multiplicity of objects that are substantially opaque to the electromagnetic radiation of interest and each of which is comparable in size to the wavelength of radiation of interest.
- 4 An optic as claimed in Claim 1 in which the membrane and the diffraction apertures are both of material which is substantially transparent to the electromagnetic radiation, but in which transmitted radiation experiences a relative phase change depending on which material it passed through, and in which the relative phase change is sufficient to introduce substantial diffraction.
5. An optic as claimed in any of Claims 1 to 4 intended for use in the soft X-ray region of the electromagnetic spectrum, which is taken to extend from 0.5 to 50 nanometers.
- 6 An optic substantially as described herein with reference to Figures 1 to 5.

-6 -

Patents Act 1977

Examiner's report to the Comptroller under
Section 17 (The Search Report)

Application number

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Relevant Technical fields

(i) UK CI (Edition K) H5R (REU, RFQ, RCD)
G2J CJ33X(ii) Int CL (Edition 5) G02B
H05G

Search Examiner

J COULES

Databases (see over)

(i) UK Patent Office

(ii) ONLINE DATABASES: WPI

Date of Search

14 JULY 1992

Documents considered relevant following a search in respect of claims

1-6

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
X	GB 591628 (SCOPHONY) - See particularly page 5 lines 24-48 and subsequent references to X-rays	1, 2
X	US 4746205 (WESTINGHOUSE) - See particularly diffraction plates 12 and 72 in Figures 4 and 5	1, 2

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Category	Identity of document and relevant passages	Relevant to claim(s)

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P: Document published on or after the declared priority date but before the filing date of the present application.

E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.

&: Member of the same patent family, corresponding document.

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